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“पुराने को छोड़ नये के तरफ”

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IS 13963-1 (1994): Chemical analysis of cadmium metal, Part 1: Determination of copper, zinc, lead and iron [MTD 9: Lead, Zinc, Cadmium, Tin, Antimony and their Alloys]



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“Knowledge is such a treasure which cannot be stolen”

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भारतीय मानक

कैडमियम धातु का रासायनिक विश्लेषण

भाग 1 ताँबा, जस्ता, सीसा तथा लोहा ज्ञात करना

Indian Standard

CHEMICAL ANALYSIS OF CADMIUM METAL

PART 1 DETERMINATION OF COPPER, ZINC, LEAD AND IRON

UDC 669.73 : 543 : 546.47.56.728.15

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BUREAU OF INDIAN STANDARDS
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NEW DELHI 110002

FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards, after the draft finalized by the methods of Chemical Analysis of Non-Ferrous Metals Sectional Committee had been approved by the Metallurgical Engineering Division Council.

IS 6344 : 1985 'Specification for cadmium metals' covers the requirements of various grades of this material. The Sectional Committee responsible for preparation of this standard decided that the methods of chemical analysis of various constituents prescribed in IS 6344 : 1985 may be covered in different parts of the standard. Accordingly this standard (Part 1) has been prepared to describe the atomic absorption spectrometric method for determination of copper, zinc, lead and iron in cadmium metal. The spectrophotometric method for determination of copper and iron and polarographic method for determination of zinc and lead have also been prescribed as alternate methods.

The Part 2 of this standard describes the spectrophotometric methods for determination of nickel, arsenic, antimony and thallium in cadmium metal.

In reporting the results of test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'.

*Indian Standard***CHEMICAL ANALYSIS OF CADMIUM METAL****PART 1 DETERMINATION OF COPPER, ZINC, LEAD AND IRON****1 SCOPE**

This standard (Part 1) describes atomic absorption spectrometric methods for determination of copper, zinc, lead and iron as the referee methods. It also describes spectrophotometric method for determination of copper and iron and polarographic method for determination of zinc and lead as alternate methods.

2 REFERENCES

The following Indian Standards are necessary adjuncts to this standard:

<i>IS No.</i>	<i>Title</i>
264 : 1976	Nitric acid (<i>second revision</i>)
265 : 1993	Hydrochloric acid (<i>fourth revision</i>)
1817 : 1961	Methods of sampling of non-ferrous metal for chemical analysis
1070 : 1992	Reagent grade water (<i>third revision</i>)

3 SAMPLING

Sample shall be drawn and prepared in accordance with the procedure laid down in IS 6344 : 1985 and IS 1817 : 1961.

4 QUALITY OF REAGENTS

Unless specified otherwise, analytical grade reagents and distilled water (*see* IS 1070 : 1992) shall be employed for the test.

5 DETERMINATION OF COPPER, ZINC LEAD AND IRON BY ATOMIC ABSORPTION SPECTROMETRIC METHOD**5.1 Outline of the Method**

The sample is decomposed by treatment with hydrochloric acid and nitric acid. The mass is evaporated to syrupy consistency and then dissolved in water. The aliquot is taken to measure the absorbance of copper, zinc, lead and cadmium against standard solution containing all the subject elements at wavelengths 324.7, 213.9, 217.9 and 248.3 nm respectively.

5.2 Apparatus

5.2.1 Any standard atomic absorption spectrophotometer with wavelength range from 200 to 900 nm is suitable (*see* Note 1).

NOTE 1 — Instruments that can read in concentration mode and with auto zero facility will be of advantage.

5.2.2 *Hollow Cathode Lamps* for Zn, Cu, Pb, and Fe.

5.3 Reagents

5.3.1 *Hydrochloric acid*, rd = 1.16 (conforming to IS 265 : 1993)

5.3.2 *Nitric Acid*, rd = 1.42 (conforming to IS 264 : 1976)

5.3.3 *Dilute Nitric Acid*, 1 percent, 50 percent (v/v)

5.3.4 *Dilute Hydrochloric Acid*, 1 percent (v/v)

5.3.5 Standard Solutions**5.3.5.1 Standard copper solution (100 ppm)**

Transfer 1.00 g of copper (purity 99.99 percent min) to a 250 ml beaker and add 15 ml of dilute nitric acid (1 : 3), warm gently until copper dissolves. Cool, add 20 ml water and boil again. Transfer the solution to 1 litre volumetric flask and make up. Mix thoroughly. Take 10 ml of this solution and dilute to 100 ml. (Dilution is done as and when required).

5.3.5.2 Standard zinc solution (100 ppm)

Dissolve 1.00 g of zinc (purity 99.99 percent min) in 20 ml dilute hydrochloric acid (1 : 4). Dilute to 1 litre. Take 10 ml of this solution and dilute to 100 ml. (Dilution is done as and when required).

5.3.5.3 Standard lead solution (100 ppm)

Dissolve 1.00 g of pure lead in minimum volume of nitric acid and dilute to 1 litre with dilute nitric acid (1 percent) or dissolve 1.598 g of lead nitrate in dilute nitric acid (1 percent) and dilute to 1 litre with dilute nitric acid (1 percent). Dilute 10 ml of this solution to 100 ml (Dilution is done as and when required).

5.3.5.4 Standard iron solution (100 ppm)

Dissolve 1.00 g of pure iron in 50 ml of dilute nitric acid (50 percent). Dilute to 1 litre with distilled water. Dilute 10 ml of this solution to 100 ml. (Dilution is done as and when required).

5.4 Standard Conditions for Determination of Zn, Cu, Pb and Fe by Atomic Absorption Spectrometer

Element	Light Source	Wavelength (nm)	Slit Width (nm)
Cu	Hollow cathode lamp	324.7	0.7
Zn	do	213.9	0.7
Pb	do	217.0	0.7
Fe	do	248.3	0.2

Recommended flame, air-acetylene, oxidizing (lean, blue).

5.5 Sample Preparation

5.5.1 Take 10 g of sample and transfer to beaker. Add 35 ml of hydrochloric acid and 10 ml of dilute nitric acid (1 : 1). When dissolution is complete, boil down the assay to a syrupy consistency. Transfer the contents to 100 ml volumetric flask, make up to volume and mix thoroughly.

5.5.2 Pipette out 20 ml each of the sample into two 100 ml standard flasks. Mark them as *B* and *C*. Make up *B* with dilute nitric acid (1 percent). To *C* add 1 ml each of standard copper, zinc, lead and iron solution (100 ppm) and make up with dilute nitric acid (1 percent).

5.5.3 Blank Solution

Prepare a reagent blank containing the same amount of reagents and spectrographically pure cadmium metal instead of sample and taken through 5.5.1 and 5.5.2. Mark the blank solution as *A*.

5.6 Determination of Copper

Align the instrument. Mount copper hollow cathode lamp, set wavelength at 324.8 nm, slit width at 0.7 nm, feed lamp current and optimise the instrument as per the instructions manual. Turn on air, acetylene and flame. Aspirate distilled water and adjust for zero absorbance. Then aspirate standard copper solution (5 ppm) and adjust the instrument to maximise absorbance (about 0.35 absorbance units).

Aspirate blank solution *A* and record absorbance if any. Also aspirate sample solution *B* and (sample + standard) solution *C*. Record absorbance in each case. An average of at least five readings is taken in each case (see Note 2).

5.6.1 Calculation

$$\text{Copper, percent by mass} = \frac{b_1 - a_1}{(c_1 - b_1)} \times 200$$

where a_1 , b_1 and c_1 are absorbances of solution *A*, *B* and *C* respectively.

NOTE 2 — The method of standard additions may be followed in case atomic absorption spectrometer can directly read in concentration mode and is provided with auto zero facility.

Real sample solution *B* in concentration mode and set the photometric reading to zero by depressing auto zero switch. Then aspirate the (sample + standard) solution *C* and set the concentration to 1 ppm (concentration of standard added in flask *C* is equal to 1.0 ppm). Read the concentration of the blank *A*. Then the absolute value of the negative reading obtained will correspond to the concentration of the element in the sample solution.

Element, percent by mass

$$= \frac{\text{concentration in } \mu\text{g/ml} \times 0.01}{\text{mass in g of sample}}$$

5.7 Determination of Zinc

Mount zinc hollow cathode lamp, set wavelength at 213.9 nm and optimise the instrument as per instructions manual. Ignite the flame. Aspirate distilled water and adjust the absorbance to zero. Record the absorbances of solutions *A*, *B* and *C*.

5.7.1 Calculation

$$\text{Zinc, percent by mass} = \frac{(b_2 - a_2)}{(c_2 - a_2)} \times 200$$

where a_2 , b_2 and c_2 are absorbances of solutions *A*, *B* and *C* respectively.

5.8 Determination of Lead

Mount the lead hollow cathode lamp, set wavelength at 217.0 nm and optimise the instrument as per the instructions manual. Ignite the flame. Aspirate distilled water and adjust the absorbance to zero. Record the absorbances of solutions *A*, *B* and *C*.

5.8.1 Calculation

$$\text{Lead, percent by mass} = \frac{(b_3 - a_3)}{(c_3 - b_3)} \times 200$$

where a_3 , b_3 , and c_3 are absorbances of solutions *A*, *B* and *C* respectively.

5.9 Determination of Iron

Mount the iron hollow cathode lamp, set wavelength at 248.3 nm and optimise instrument as per the instructions manual. Ignite the flame. Aspirate distilled water and adjust the absorbance to zero. Record the absorbances of solutions *A*, *B* and *C*.

5.9.1 Calculation

$$\text{Lead, percent by mass} = \frac{(b_4 - a_4)}{(c_4 - b_4)} \times 200$$

where a_4 , b_4 and c_4 are absorbances of solutions *A*, *B* and *C* respectively.

6 DETERMINATION OF COPPER BY THE SPECTROPHOTOMETRIC METHOD

6.1 Outline of the Method

Sample is dissolved in hydrochloric acid and nitric acid. Copper present is reduced to the cuprous state with hydroxylamine hydrochloride, complexed with bathocuproin reagent and estimated spectrophotometrically at 457 nm.

6.2 Reagents

6.2.1 Hydrochloric Acid, rd = 1.16 (conforming to IS 265 : 1993)

6.2.2 Nitric Acid, rd = 1.42 (conforming to IS 264 : 1976)

6.2.3 Dilute Sulphuric Acid, 50 percent (v/v).

6.2.4 Hydroxylamine Hydrochloride, 10 percent (m/v) in hydrochloric acid 1 M.

6.2.5 Tartaric Acid, 10 percent (m/v) in water.

6.2.6 Bathocuproin Reagent, 0.02 percent.

Dissolve 0.2 g of bathocuproin in 1 litre of iso amyl alcohol.

6.2.7 Ammonia Solution, 5 percent (v/v).

6.2.8 Standard Copper Solution (1 ml = 5 µg Cu)

Take 0.1 g of pure copper (purity 99.99 percent min) and dissolve in 5 ml of dilute nitric acid (1 : 1). Boil the solution and transfer to 1 litre volumetric flask. Make up to the mark. This solution contains 100 µg of copper per ml. Dilute 5 ml of this solution to 100 ml. The final solution contains 5 µg of copper per ml.

6.3 Procedure

6.3.1 Take 5 ml of the sample solution prepared under 5.5 in 150 ml breaker. Add 5 ml each of hydroxylamine hydrochloride and tartaric acid solutions and take the assay to boil. Cool and adjust the pH to about 5.5 by addition of dilute ammonia solution (5 percent).

6.3.2 Transfer to 150 ml separating funnel. Add 20 ml bathocuproin reagent. Shake well for one minute. Allow layers to separate. Drain off the aqueous layer and read the absorbance of the reagent layer in a spectrophotometer at 457 nm. A blank taken through all the stages of the procedure is used as a reference solution.

6.3.3 Convert absorbance reading of the sample solution to mg of copper from the calibration curve and calculate the percentage of copper.

6.3.4 Calibration Curve

Take 0, 1, 2, 3, 4 and 5 ml of the standard copper solution (6.2.8) in a series of 150 ml beakers. Add 5 ml each of hydroxylamine hydrochloride and tartaric acid and proceed as in 6.3.1 and 6.3.2. Record the absorbance of standard solutions and draw the calibration curve.

6.3.5 Calculation

$$\text{Copper, percent by mass} = \frac{A}{B} \times 100$$

where

A = mass in mg, of copper present in sample; and

B = mass in mg, of sample taken.

7 DETERMINATION OF IRON BY SPECTROPHOTOMETRIC METHOD

7.1 Outline of the Method

Sample is brought into solution with dilute nitric acid. Nitric acid is then removed by baking with hydrochloric acid and the mass is taken into solution in hydrochloric acid. Cadmium is removed by hydrogen sulphide. After removal of hydrogen sulphide, iron is reduced to divalent state by the addition of hydroxylamine hydrochloride. The colour is then developed by the addition of 1:10 phenanthroline and the absorbance of the solution measured at 510 nm.

7.2 Reagents

7.2.1 Hydrochloric Acid, rd = 1.16 (conforming to IS 265 : 1993).

7.2.2 Nitric Acid, rd = 1.42 (conforming to IS 264 : 1976).

7.2.3 Hydroxylamine Hydrochloride Solution, 100 g/litre.

Dissolve 10 g of hydroxylamine hydrochloride ($\text{HONH}_2 \cdot \text{HCl}$) in about 60 ml of water and dilute to 100 ml.

7.2.4 Standard Iron Solution (1 ml = 0.01 mg). Dissolve 0.1000 g of iron 10 ml of hydrochloric acid (1 : 1) and 1 ml of bromine water. Boil until excess bromine is removed. Add 200 ml of hydrochloric acid, cool and dilute to 1 litre. Dilute 100 ml of this solution to 1 litre.

7.2.5 Ortho Phenanthroline Solution (3 g/l)

Dissolve 0.9 g of o-phenanthroline in 30 ml of methanol and dilute to 300 ml with water.

7.2.6 Iron Sulphide, solid.

7.3 Calibration Curve

Transfer 0 (reagent blank) 2, 4, 6, 8 and 10 ml respectively of standard iron solution to each of six 100 ml volumetric flasks. Dilute to 80 ml with distilled water. Add 5 ml of hydroxylamine hydrochloride solution, shake and then add 5 ml of reagent i.e. 1 : 10 phenanthroline solution to develop (red colour). After 15 minute record the absorbance of each solution at 516 nm. Draw the calibration curve by plotting micrograms of iron as function of absorbance.

7.4 Procedure

Take 1 g of the sample. Dissolve in 10 ml of dilute nitric acid (1 : 1). After the metal is brought into solution, add hydrochloric acid and evaporate to paste. Again add 10 ml hydrochloric acid. Boil and dilute to 100 ml. Pass hydrogen sulphide gas to remove cadmium (yellow precipitate). Filter off the precipitate and boil the solution with 20 ml hydrochloric

acid to remove hydrogen sulphide from the solution (*see* Note 3). When hydrogen sulphide is removed; dilute the solution to 80 ml. Add 5 ml hydroxylamine hydrochloride, 5 ml reagent and adjust the pH from 3 to 4 by adding ammonium hydroxide or dilute hydrochloric acid. Wait for 15 minutes to stabilize the colour and measure the absorbance at 510 nm against reagent blank in spectrophotometer using 1 cm cell. Convert the absorbance reading to μg of iron from the calibration curve and calculate the percentage of iron.

NOTE 3 — In case iron concentration in the sample is more, take suitable aliquot and proceed.

7.5 Calculation

$$\text{Iron, percent by mass} = \frac{M}{m \times 10^6}$$

where

M = mass in μg , of the iron in the sample;
and

m = mass in g, of the sample taken.